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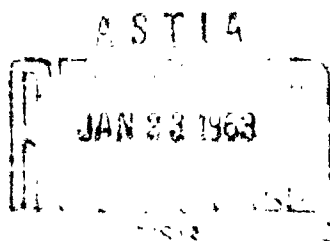
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THE PREPARATION OF A POWDER-METALLURGY COBALT-  
TUNGSTEN ALLOY FOR INSTRUMENT MAKING

By

A. V. Savin, Yu. A. Eyduk

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# UNEDITED ROUGH DRAFT TRANSLATION

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# THE PREPARATION OF A POWDER-METALLURGY COBALT-TUNGSTEN

## ALLOY FOR INSTRUMENT MAKING

A. V. Savin, Yu. A. Eyduk

The authors have tested various regimes for the preparation of a cobalt-tungsten alloy by powder-metallurgy methods. Moreover, the fundamental parameters affecting the preparation of a dense single-phase alloy with a reduced gas content have been found.

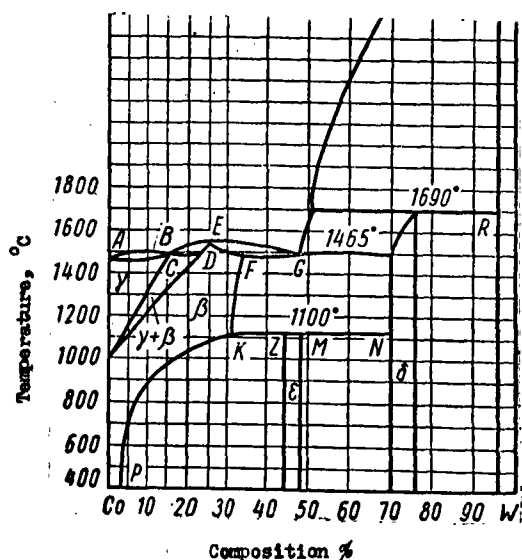


Fig. 1. Phase diagram of cobalt-tungsten system

An alloy of cobalt with tungsten possesses, after appropriate thermomechanical treatment, improved strength and wear-resistance properties. Since it is at the same time a corrosion-resistant material, this alloy has been used in place

of alloy steel, e.g., in the building of vibration-proof axles in measuring instruments.

The phase diagram of a cobalt-tungsten system is given in Sikes' paper [1]. As is apparent from the diagram (Fig. 1), the cobalt dissolves about 35% of the tungsten at  $1465^{\circ}$  (i.e., at the eutectic temperature). At  $350^{\circ}$  the cobalt dissolves a total of only 3% of the tungsten. In the case of an alloy containing 25% tungsten a  $\beta$ -solid solution exists at temperatures above  $1075^{\circ}$ , while at temperatures below  $1075^{\circ}$  the alloy consists of two phases:  $\beta$  and  $\epsilon$ . The separation of the  $\epsilon$ -phase in a supersaturated solution leads to dispersion hardening of the alloy at temperatures above  $500^{\circ}$ .

A powder-metallurgy method of preparing the alloy from cobalt and tungsten powders (75% Co and 25% W by weight) was considered in St. Stolarz' paper [2].

Cobalt in powder form was obtained by reduction at  $700^{\circ}$  for three hours in a hydrogen atmosphere. Tungsten powder was prepared by the oxidation of tungsten scrap at  $1000^{\circ}$  and subsequent reduction in a hydrogen stream at the same temperature. After mixing in a porcelain mill the charge was completely reduced in a hydrogen atmosphere and then pulverized and screened. The molds were pressed from the mixture thus obtained at a specific pressure of 2 metric tons per  $\text{cm}^2$ . The author points out that flaking appears when the molding pressure is raised above 3 metric tons per  $\text{cm}^2$ . The sintering temperature of the cobalt-tungsten molds was chosen as  $1400^{\circ}$  with a ten-hour holding. As a result, metallic molds with a homogeneous structure were obtained, but with an extremely low density— $8.67 \text{ g/cm}^3$ . After the molds were pressure-treated, their density increased and approached the theoretical, i.e.,  $10.28 \text{ g/cm}^3$ .

As the initial raw material for the preparation of the metallic tungsten powder the authors of the article used ammonium paratungstate of the "Pobedit" plant, as well as tungsten anhydride from the hard-alloys plant of the Sverdlovsk Council of National Economy. The ammonium paratungstate was roasted in a muffle furnace at

800°, until tungsten anhydride was obtained. The reduction of the tungsten anhydride to tungsten was carried out in a hydrogen stream in two stages in a two-tube laboratory direct-heating furnace with the inner diameter of the tubes being 51 mm and the length of the heating zone being 1500 mm.

Nickel boats 30 by 200 mm were used for the reduction. Fifty grams of tungsten anhydride were loaded into each boat.

The reduction regimes are given in Table 1.

After reduction, the tungsten powder was screened by hand through a No. 0.112-0.1 (130-150 mesh) sieve and placed in a glass jar with a ground-in stopper, in order to preserve it.

TABLE 1

Regimes of the Reduction of Tungsten

Anhydride to Metallic Tungsten			
Reduction stages	Reduction temperature °C	Rate of advance of boat, mm/min	Amount of hydrogen supplied through tube, liters/hr
I	650	13.3	800—1000
II	800	10.0	800—1000

TABLE 2

Characteristics of the Metallic Tungsten Powder Obtained

Absorption of methanol, wt/g	Amount of oxygen, %	Pour weight, g/cm <sup>3</sup>	Comment
0,3—0,6	0,2—0,3	0,9—1,0	From ammonium paratungstate from the "Pobedit" plant.
0,3—0,6	0,2—0,3	1,0—1,1	From tungsten anhydride from the hard-alloys plant of the Sverdlovsk Council of National Economy.

Cobalt trioxide  $\text{Co}_2\text{O}_3$  was reduced to metallic cobalt in the same furnace in iron boats 30 by 400 mm according to the regime given below:

Weight of load in boat, g .....	120
Reduction temperature, $^{\circ}\text{C}$ .....	580
Rate of advance, mm/min .....	13.3
Amount of hydrogen fed through tube, liters/hr .....	1000

The powdered cobalt thus obtained was screened freely by hand through a No. 0.112 (130 mesh) sieve and poured into a glass jar with a ground-in stopper.

The characteristics of the cobalt obtained are given below:

Amount of oxygen, % .....	0.2—0.5
Adsorption of methanol, mg/g .....	0.4—0.5
Pour weight, $\text{g}/\text{cm}^3$ .....	0.6—0.7

The charge, which consisted of 75% Co and 25% W by weight, was prepared by mixing the cobalt and tungsten powders in a 5 liter porcelain mill 180 mm in diameter. Porcelain balls 25 mm in diameter served as the mixing bodies. The ratio of the weight of the balls to the weight of the charge was 1:2. When the weight of the balls was increased, cold-hardened cobalt in the form of bright scales was obtained in the charge. The mixture thus obtained was pressed into molds 10 x 10 x 400 mm in a sectional steel die. The specific molding pressure chosen was 3 metric tons per  $\text{cm}^2$ .

The specimens were sintered in an Alundum furnace with a molybdenum winding in two stages with different variations of temperature and time in a hydrogen atmosphere with a moisture content of 10-12 and a "dew point" of  $25^{\circ}$ .

For the high temperature sintering the specimens were placed in a molybdenum boat on a corrax burden. During the sintering of the cobalt-tungsten molds their density was brought up to the compact metallic state. Also, at certain sintering regimes complete homogenization with respect to composition was achieved, and a single-phase structure in the  $\beta$ -solid solution was obtained.



Listed below are the changes in the density of the alloy specimens as a function of the temperature of the first sintering in the interval from 800° to 1100°:

Sintering temperature, °C .....	800	900	1000	1100
Specific gravity, g/cm <sup>3</sup> .....	5.42	6.06	7.80	8.59

At the preliminary sintering temperature (800°) there was practically no shrinkage in the specimens. With an increase in temperature into the range from 900° to 1100° the density of the specimens increased noticeably.

Data pertaining to the effect of the sintering regime on the microstructure and density of the cobalt-tungsten alloy are given in Table 3.

It is apparent from Table 3 that the appearance of foreign inclusions in the form of isolated impregnations of the other phase (cf. Fig. 5) in the cobalt-tungsten alloy is connected with the temperature regime of the sintering.

Thus, a first sintering at 800° (series I) did not give foreign inclusions in the finally sintered alloy. When the temperature of the first sintering equaled 1100° (series II) with the same regimes for the second sintering, the cobalt-tungsten alloys, as a rule, had foreign inclusions. The reason for the appearance of these inclusions and the question of their composition remain unclear.

In specimens that were heated immediately to a temperature of 1300-1350° without a first sintering (series III), the process of homogenization took place completely, but in this case the specimens had an increased porosity of approximately 2-4% by contrast to specimens which had undergone a first sintering and whose porosity amounted to 0.2-1.5%.

The results regarding the influence of the duration of the first sintering on the microstructure and density of a cobalt-tungsten alloy, when the first sintering is carried out at 800° and the second sintering at various temperatures, are presented in Table 4.

It is apparent from the table that an increase in the time of the low-temperature

first sintering of up to 3 hours does not affect the homogenization and density of the alloy during the final second sintering.

The microstructure and density of the alloy specimens were determined by metallographic investigation. Typical structures of specimens obtained using different sintering regimes are presented in Figs. 2-5.

TABLE 3

The Effect of the Sintering Regime on the Microstructure  
and Density of Cobalt-Tungsten Alloy

Series No.	Regime of first sintering		Regime of second sintering		Specific gravity, g/cm <sup>3</sup>	Metallographic study	
	Temperature, °C	Time, hrs.	Temperature, °C	Time, hrs.		Presence of inclusions	
I	800	2	1250	2	10.18	Yes	3—6
	800	2	1250	4	10.24	No	6—9
	800	2	1300	2	10.18	Yes	12—18
	800	2	1300	4	10.27	No	12—18
	800	2	1350	2	10.25	"	25—40
	800	2	1350	4	10.28	"	40—50
II	1100	2	1250	2	10.18	Yes	3—6
	1100	2	1250	4	10.26	"	6—9
	1100	2	1300	2	10.13	"	6—12
	1100	2	1300	4	10.26	"	25—40
	1100	2	1350	2	10.26	"	20—30
	1100	2	1350	4	10.28	"	25—40
III	—	—	1250	2	10.0	Yes	3—6
	—	—	1250	4	10.16	"	3—6
	—	—	1300	2	10.12	"	6—9
	—	—	1300	4	10.19	"	6—9
	—	—	1350	2	10.12	"	25—40
	—	—	1350	4	10.20	"	12—8

The sintered specimens of cobalt-tungsten alloys were analyzed for gas content. Figure 6 shows how the gas content in the initial charge and the moisture content in the hydrogen atmosphere in the furnace affect the gas content in the sintered alloy specimens as a function of the sintering duration. It is apparent from the graph that the less gas contained in the initial cobalt and tungsten powder (or charge), the less the gas content in the alloy for the same regimes of sintering. Also, a degasification of the cobalt-tungsten alloy occurs in the second sintering as the moisture content in the hydrogen atmosphere in the furnace is decreased and the holding time is increased.

The process of degasification takes place in the cobalt-tungsten material mainly

during the second final sintering. The amount of gas decreases by a factor of ten or more during the final sintering process. No noticeable degasification takes place in the first preliminary sintering. The amount of gas lies in the same range (about 0.3% to 0.5% by weight) after the first sintering, as it did in the initial cobalt and tungsten powders.

On the basis of the results obtained it is possible to draw the following conclusions regarding the conditions needed in order to prepare the most compact single-phase alloy from cobalt and tungsten powders with the above-stated purity and granularity.

1. The oxygen content in the initial powders should not exceed 0.3% in the tungsten and 0.5% in the cobalt.
2. In order to reduce the residual-gas content, the sintering process must be carried out in a hydrogen atmosphere with a moisture content for which the "dew point" is no higher than 12°.
3. For complete homogenization of the alloy and at the same time high compactness the following regime will be optimal:  
first sintering at 800° for 1-2 hrs.  
second sintering at 1300-1350° for 4 hrs.

TABLE 4  
Influence of the Time of the First Sintering on the Microstructure  
and Density of the Cobalt-tungsten Alloy for a First Sintering Temperature of 800° and Various Second Sintering Temperatures

Time of first sintering hrs.	Second sintering temperature with a 2-hr. holding	Specific gravity, g/cm <sup>3</sup>	Metallographic investigation	
			Presence of inclusions	Bulk of grains,
1	1250	10.1	No	3-6
3	1250	10.08	"	3-6
1	1300	10.15	"	6-9
3	1300	10.13	"	6-9
1	1350	10.19	"	6-9
3	1350	10.21	"	6-12



Fig. 2. Single-phase structure of  $\beta$ -solid solution. First sintering at  $800^{\circ}$ ; sintering time: 2 hrs. Second sintering at  $1350^{\circ}$ ; sintering time: 2 hrs. Size from  $6-9\mu$ . x 400. Etched in a mixture of hydrogen peroxide (10%) and sulfuric acid (50:1 concentration)

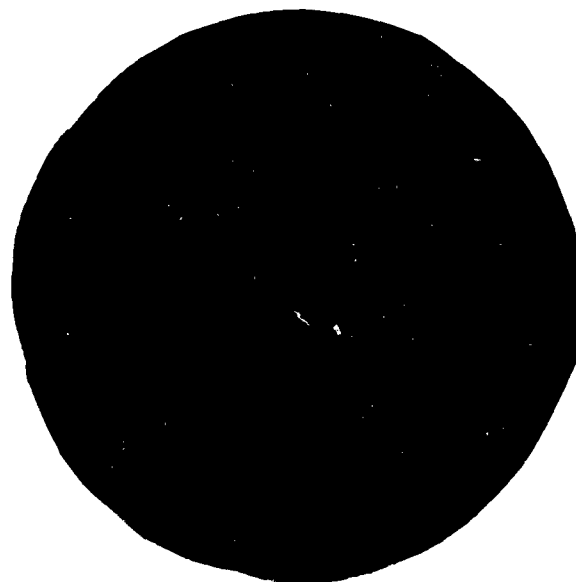


Fig. 3. Single-phase structure of  $\beta$ -solid solution. First sintering at  $800^{\circ}$ ; sintering time 2 hrs. Second sintering at  $1350^{\circ}$ ; sintering time 4 hrs. Grain size from 6 to  $18\mu$ . x 400. Etched as in Fig. 2.



Fig. 4. Porosity of cobalt-tungsten alloy. x 90.

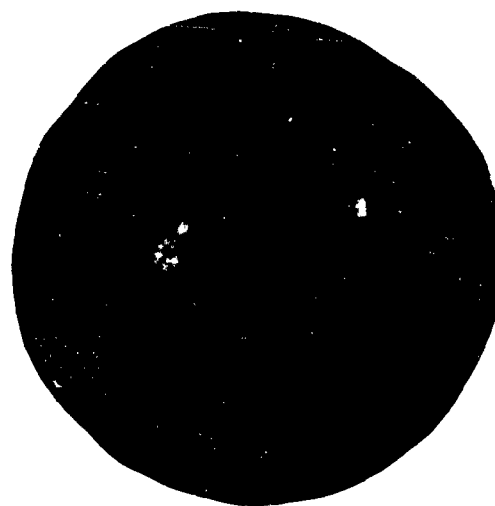


Fig. 5. Alloy with a small number of foreign inclusions. First sintering at  $1100^{\circ}$ ; sintering time: 2 hrs. Second sintering at  $1300^{\circ}$ ; sintering time: 2 hrs. x 400. Etched in a saturated solution of cupric chloride in concentrated nitric acid.

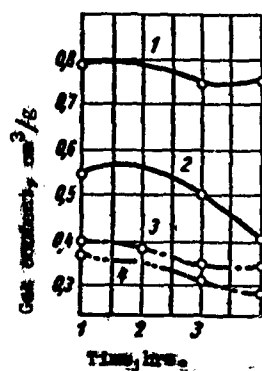


Fig. 6. Measurement of the gas content in cobalt-tungsten molds as a function of the variation in the sintering time for hydrogen atmospheres with various moisture contents; sintering temperature:  $1350^{\circ}$ : 1) about 1.0%  $O_2$  by weight in cobalt-tungsten charge before sintering; 2) about 0.5%  $O_2$  by weight in cobalt-tungsten charge before sintering.

#### REFERENCES

1. Sikes. Trans. of the American Society for Steel Treating, V. XXI, 1933, p. 5.
2. St. Stolarz. Metal and Production of Cobaltfug Sten alloy, 55, IX-X. 1953, p. 298-302.

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